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SUMMARY

Friction and wear studies were conducted with four material combinations run submerged in liquid oxygen and in liquid fluorine to determine their potential as dynamic seal components for fluorine turbopump applications. The friction and wear experiments were conducted with a 3/16-inch-radius hemispherically tipped rider sliding in a circumferential path on the flat surface of a rotating $2\frac{1}{2}$-inch-diameter disk.

The seals used in this investigation had a flame-sprayed Al_2O_3 nosepiece (0.006 to 0.008 in. thick) and were run against a mating disk of TiC cermet or a fused coating of CaF_2 + LiF + NiF_2 on Al_2O_3 submerged in liquid fluorine.

Results indicated that the presence of a fluoride film, either as an applied fused coating (CaF_2 + LiF + NiF_2) or as a film formed during sliding (NiF_2 on the TiC cermet or possibly aluminum fluoride (AlF_3) on Al_2O_3) in liquid fluorine, was beneficial in reducing the friction and wear of the Al_2O_3 riders. The seal experiments in liquid fluorine showed that flame-sprayed Al_2O_3 sliding against the TiC cermet or a fused coating of CaF_2 + LiF + NiF_2 on Al_2O_3 are potential seal materials for fluorine turbopump applications.

INTRODUCTION

The potential use of liquid fluorine as the oxidizer for high-thrust rocket engines imposes critical problems for sliding contact seals in turbopumps. To date little information is available to aid in the selection of sliding contact seal materials for use in liquid fluorine. Materials that have been used successfully in seals for liquid hydrogen and liquid oxygen applications have also been used in liquid fluorine with some problems. The successes that have been achieved can generally be attributed to the use of a sweep gas across the seal face, which reduces contact of the seal materials with fluorine (ref. 1).

Instances of violent explosions have occurred, however, with both oxygen and fluorine where carbons have been used and the ignition source has been traced to the seal areas. Compatibility studies have shown that "amorphous" carbon will react violently with very low concentrations of fluorine in nitrogen and that graphitic carbon, after prolonged soaking, will also react violently (refs. 2 to 4). Applications, therefore, should be avoided where contact of the carbon with liquid fluorine is possible. Material combinations must be selected that will be compatible with fluorine during sliding contact and will provide the low wear and friction necessary for dynamic seals. Such selections can be based on well-known chemical thermodynamic principles and on experimental reaction rate and compatibility studies (refs. 5 to 7). Reactive-gas lubrication studies (ref. 8) showed that controlled reaction rates (i.e., limiting the degree of corrosion) were useful in providing effective lubrication. It is possible, therefore, that materials that have low reaction rates or are able to resist excessive corrosion with fluorine may also form beneficial lubricating films.

The following investigation was accomplished in three phases: (1) friction and wear studies in liquid oxygen of material combinations expected to be chemically stable in fluorine, (2) friction and wear studies of these combinations in liquid fluorine, and (3) full-scale seal studies of these combinations in liquid fluorine at surface speeds and loads representative of a pump application.

MATERIAL SELECTION

The solid materials used in this investigation were selected primarily for their resistance to fluorine attack. The TiC cermet and Al_2O_3 were selected because of hardness, resistance to wear, and chemical stability in fluorine. The nickel-chrome alloy that contains approximately 55 percent nickel was selected as the substrate for the fused fluoride coating (62 BaF_2 + 38 CaF_2, eutectic) because of its hardness and the tight bond formed between this coating and nickel-chrome alloys. Anticipated shear properties of passivation films in sliding contact were also important in the selection of materials.

The two fused fluoride coatings (76 CaF_2 + 23 LiF + 1 NiF_2 and 62 BaF_2 + 38 CaF_2) were selected because they are very stable compounds that would not be chemically affected by liquid fluorine. These fused fluoride coatings have also shown considerable promise as solid lubricants in other extreme environments (ref. 9).

APPARATUS AND PROCEDURE

The apparatus used in the first two phases of this investigation is shown schematically in figure 1. The basic elements are a rotating disk specimen ($2\frac{1}{2}$-in. diam.) and a stationary hemispherically tipped rider specimen (3/16-in. rad.) in sliding contact with the disk (see inset). The disk is rotated by a variable speed electric motor through a gearbox speed increaser coupled to the specimen shaft. Disk speed is monitored by a magnetic pickup whose output is fed into a digital readout instrument.

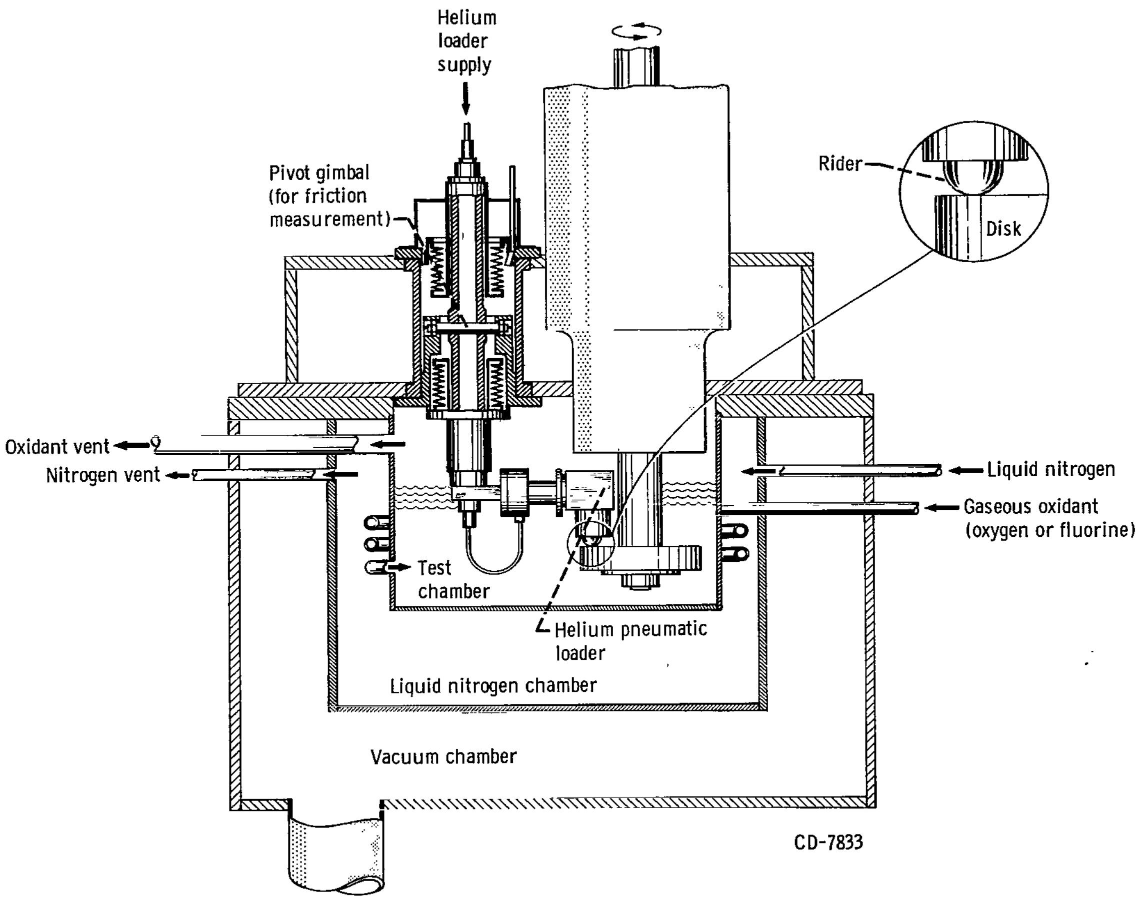


Figure 1. - Liquid oxidant friction apparatus.

The rider specimen is loaded against the disk by a pneumatically operated piston on the end of a gimbal mounted arm. Through this arm pressurizing gas (dry helium) is supplied. The arm is linked to a strain gage assembly for measurement of frictional force.

The apparatus consists of two sections (lower and upper) that may be separated for changing specimens. The lower section consists of four separate chambers. The inner chamber, or test chamber, is located within a "jacket" that is filled with liquid nitrogen for cooling and condensing the test gas. This cooling jacket is surrounded by a vacuum chamber to reduce the boiloff rate of the cooling and test fluids. The outermost or spill chamber (not shown in fig. 1) surrounds the three chambers and would provide safe operation if excess amounts of the test fluid should escape. The upper section is a vacuum jacket through which pass the disk specimen drive shaft housing and the rider specimen arm assembly. A series of face seals are used to prevent leakage of the oxidant along the drive shaft. Located in the upper half of the bearing housing are two carbon face seals used primarily to prevent leakage of the cooling oil. Within the housing that extends down into the test chamber is a double-face seal that is purged with helium set at a pressure slightly higher than the test chamber pressure. The upper half of this double seal consists of graphite running against a nickel-chrome alloy, and the lower half consists of Al_2O_3 running against a fused fluoride coating (BaF_2 + CaF_2) on the nickel-chrome alloy. Flexible bellows are used to seal the load beam assembly. Fluorinated hydrocarbon oils are used to lubricate the gearbox and the support bearings in the shaft housing to minimize the possibility of reaction of the oil with the test fluid in case the shaft seals fail.

The test gas, oxygen or fluorine, passes through a coil within the cooling jacket before entering the test chamber near the bottom. In these tests the test fluid is initially in the gaseous state; the coil, at liquid nitrogen temperature, condenses the gas into the liquid state. Excess liquid and vapor are vented by a line leaving from a point near the top of the chamber. The liquid level within the test chamber is monitored by a capacitance probe.

The cooling jacket is filled with liquid nitrogen and vented by two diametrically opposite lines. The coolant level is monitored with a carbon resistance probe.

After the desired liquid level was attained, the disk specimen was brought up to speed and the normal load was applied. When possible, experiment duration was 1 hour. The frictional force was measured continuously by resistance strain gages mounted on a dynamometer ring whose output was fed into a recording potentiometer. After completion of a run, rider wear was obtained by measuring the wear scar and calculating the wear volume.

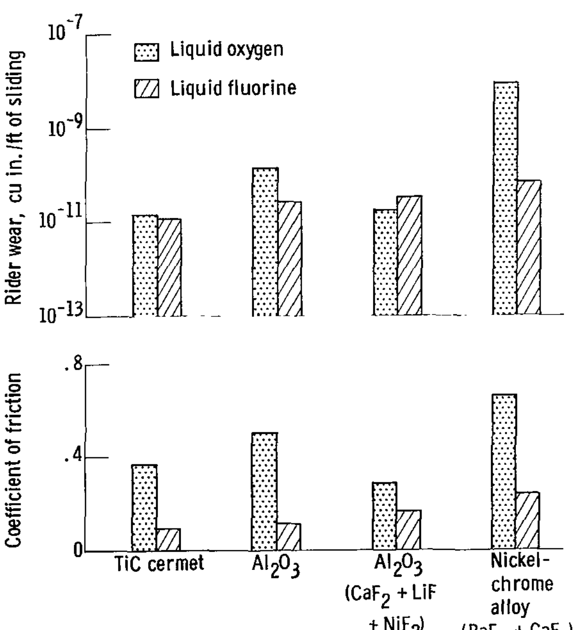


Figure 2. - Friction and wear of aluminum oxide riders sliding on various materials in liquid oxygen and liquid fluorine. Sliding velocity, 2300 feet per minute; load, 1000 grams.

For the seal experiments a housing adapter was made that contained the test seal. A 1/16-inch purge line was connected to the housing adapter, which maintained a 2-pound-per-square-inch pressure differential across the test seal.

SPECIMEN PREPARATION

For the friction and wear experiments, the solid Al_2O_3 riders and disks were scrubbed with levigated alumina, rinsed with distilled water, rinsed with alcohol, dried, and then rinsed with a solvent (1,1,1 trichloroethane) just prior to testing. The TiC cermet disk specimens were also cleaned in this manner. The fused fluoride coatings were prepared in a hydrogen atmosphere furnace at 1900° F, stored in a dessicator, and finally rinsed with a solvent (1,1,1 trichloroethane) before being placed in the friction apparatus.

For the fluorine seal experiments, the mating disk specimens were cleaned and prepared as described previously. The experimental seal assemblies were previously cleaned and passivated (exposed to fluorine gas to form a "passive" surface film). Upon assembly the flame-sprayed Al_2O_3 nosepieces were wiped with a soft cloth saturated with the solvent.

RESULTS AND DISCUSSION

Friction and Wear Experiments

Data were obtained in liquid oxygen and in liquid fluorine with Al_2O_3 riders sliding on four disk materials: (1) nickel-bonded TiC cermet, (2) solid Al_2O_3, (3) 76 CaF_2 - 23 LiF - 1 NiF_2 film on Al_2O_3 (1 NiF_2 was used as a coloring agent in order to differentiate between the fluoride coating and the Al_2O_3 substrate), and (4) 62 BaF_2 - 38 CaF_2 (eutectic) film on a nickel-chrome alloy. The friction and wear results are presented in table I and figure 2; these results are representative of a number of runs.

TABLE I. - EXPERIMENTAL DATA FOR FRICTION AND WEAR OF MATERIALS IN LIQUID OXYGEN AND LIQUID FLUORINE

[Riders, aluminum oxide; sliding velocity, 2300 feet per minute; load, 1000 grams.]

Disk material composition	Coating composition and thickness	Test liquid	Total run time, min	Rider wear rate, cu in./ft of sliding	Disk substrate wear rate, cu in./ft of sliding	Friction coefficient, f	Remarks
TiC cermet (52 Ti, 5 Mo, 25 Ni, 4.5 Cb, 13.2 C, 0.3 Ta)	None	Oxygen	60	0.015×10^{-9}	1.180×10^{-9}	0.37	Friction erratic; polished wear scars
	None	Fluorine	[a]23	0.012×10^{-9}	0.120×10^{-9}	<0.10	Friction steady; surface reaction film NiF_2 identified
Al_2O_3	None	Oxygen	60	0.140×10^{-9}	Negligible	0.50	Friction erratic; polished wear scars
	None	Fluorine	60	0.028×10^{-9}	Negligible	0.12	Friction steady; polished wear scars
Al_2O_3	76 CaF_2, 23 LiF, 1 NiF_2 0.0025 in.	Oxygen	62.5	0.015×10^{-9}	Negligible	[b]0.29	Friction steady; film failed at approximately 61.5 min
	76 CaF_2, 23 LiF, 1 NiF_2 0.003 in.	Fluorine	[a]45	0.033×10^{-9}	Negligible	0.17	Friction steady
Nickel-chrome alloy (53 Ni, 3.2 Ti, 19 Cr, 12 Fe, 11 Co, 1.6 Al, 10 Mo)	62 BaF_2, 38 CaF_2 0.0025 in.	Oxygen	59	8.300×10^{-9}	Excessive	0.66	Film failed immediately
	62 BaF_2, 38 CaF_2 0.002 in.	Fluorine	[a]20	0.076×10^{-9}	0.011×10^{-9}	0.24	Friction steady

[a]Test terminated because of low liquid fluorine level.

[b]Coefficient of friction after film failure, 0.50.

The results obtained indicate that liquid fluorine is potentially a better boundary lubricating media than liquid oxygen for the material combinations selected for this investigation. The low coefficients of friction in fluorine are attributed to the formation of beneficial metal fluoride films on the mating surfaces. The rider wear rates, cubic inches per foot of sliding, are in general of the same order of magnitude with the exception of the BaF_2 + CaF_2 eutectic coating on the nickel-chrome alloy run in liquid oxygen, which is approximately three hundred times greater. Resistance to oxidation by liquid oxygen and to fluoridation by liquid fluorine as well as hardness of the substrate materials are thought to be responsible for the low rider wear in these experiments. It appears that the corrosive and abrasive wear that could be expected in the highly corrosive environments were minimized by the careful selection of materials.

Previously reported liquid oxygen studies (refs. 10 and 11) showed that the presence of a proper oxide film on one of the mating surfaces was beneficial in effecting good lubrication. In this study it appears that the presence of a fluoride film, either an applied fused fluoride film in liquid oxygen or a fluoride film formed during sliding in liquid fluorine, is also beneficial in reducing wear and friction.

Friction and Wear with Surface Reaction Films

The results for Al_2O_3 sliding against either the TiC cermet or Al_2O_3 show a reduction in rider wear, friction coefficient, and disk wear in liquid fluorine as compared with liquid oxygen (table II and fig. 2). Photographs of

TABLE II. - EXPERIMENTAL SEAL DATA IN LIQUID FLUORINE

[Nosepiece, flame-sprayed aluminum oxide; sliding velocity, 2300 feet per minute; pressure difference, 2 lb/sq in.]

Disk material	Coating composition and thickness	Face load,[a] lb	Total run time, min	Nosepiece wear, cu in./ft of sliding	Disk substrate wear rate, cu in./ft of sliding	Remarks
TiC cermet	None	21	20	28.00×10^{-9}	7.000×10^{-9}	Surface speed, 80 ft/min
	None	15	40	6.85×10^{-9}	0.152×10^{-9}	Surface reaction; film NiF_2 identified
Al_2O_3	76 CaF_2, 23 LiF, 1 NiF_2 0.0015 in.	15	60	$<0.80\times10^{-9}$	Negligible	Trace amounts of applied fluoride film detected on wear track

[a]Calculated load at cryogenic temperature taking into account differential thermal contraction of assembly.

these two material combinations after running in oxygen and in fluorine can be seen in figures 3 and 4. Of the two sliding combinations, Al_2O_3 sliding against Al_2O_3 showed the greatest improvement in that rider wear was reduced by a factor of 5 and the coefficient of friction was reduced from 0.50 in oxygen to 0.12 in fluorine. While the combination of Al_2O_3 sliding on the TiC cermet shows no appreciable change in rider wear, the friction coefficient is reduced from 0.37 in oxygen to 0.10 in fluorine. It is important to note that continuous friction traces recorded during these runs showed that sliding was very smooth and steady in liquid fluorine as compared with the liquid oxygen, in which sliding was very rough and friction coefficient varied as much as ±0.05.

X-ray diffractions of the disk specimen wear tracks were taken after runs in the test fluids to determine whether a reaction film had formed on the materials. Nickel fluoride was identified as being present on the wear track

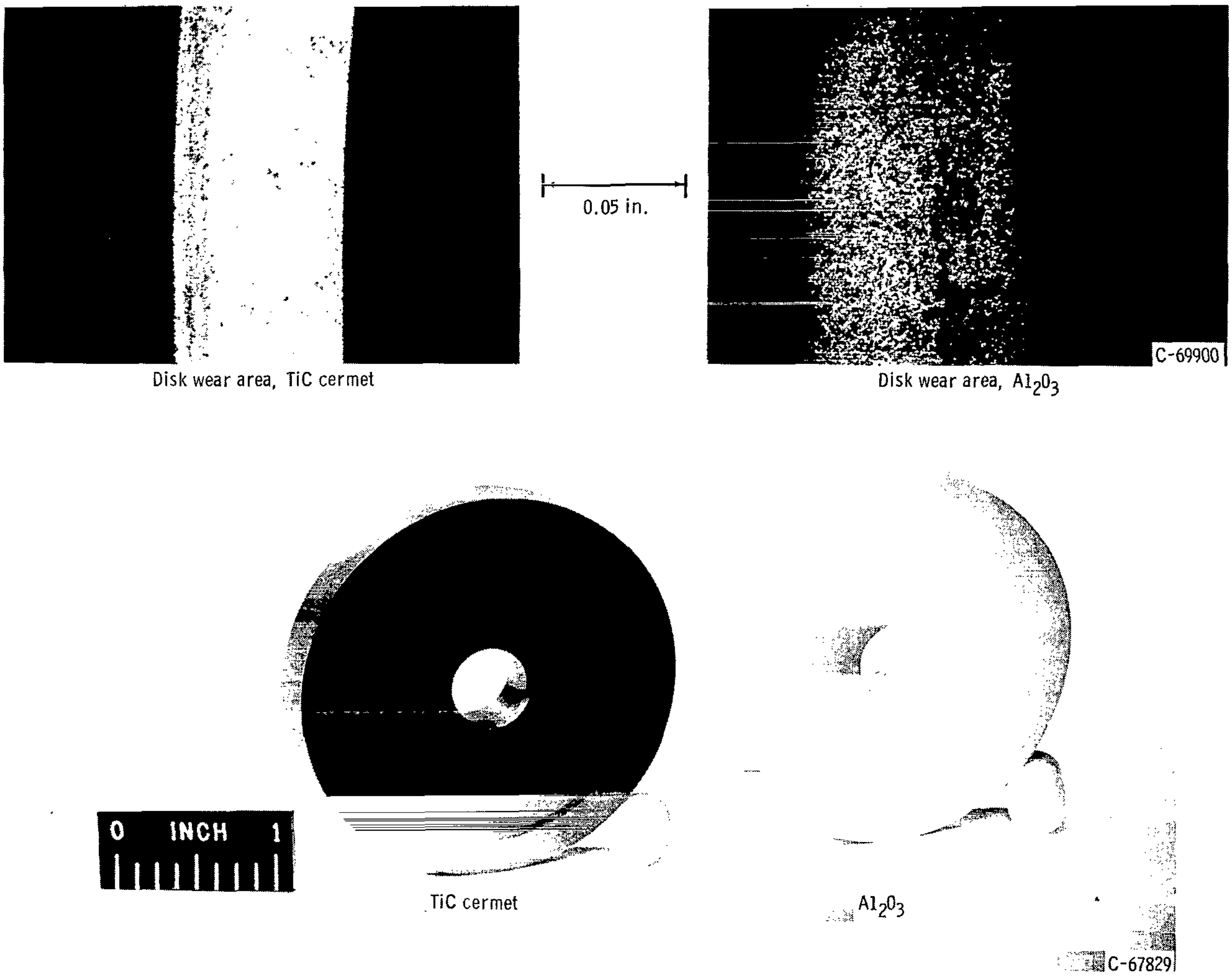
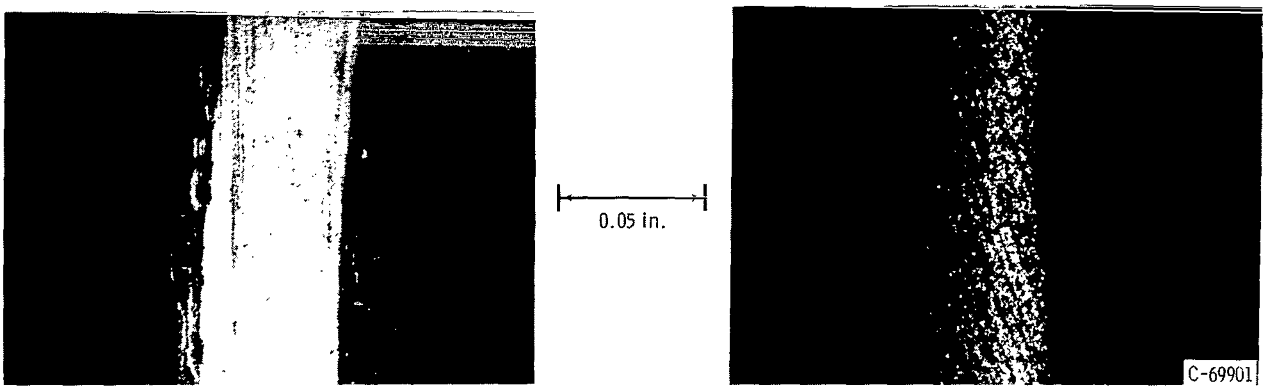


Figure 3. - Aluminum oxide riders and uncoated titanium carbide cermet and aluminum oxide disk specimens run submerged in liquid oxygen. Sliding velocity, 2300 feet per minute; load, 1000 grams.



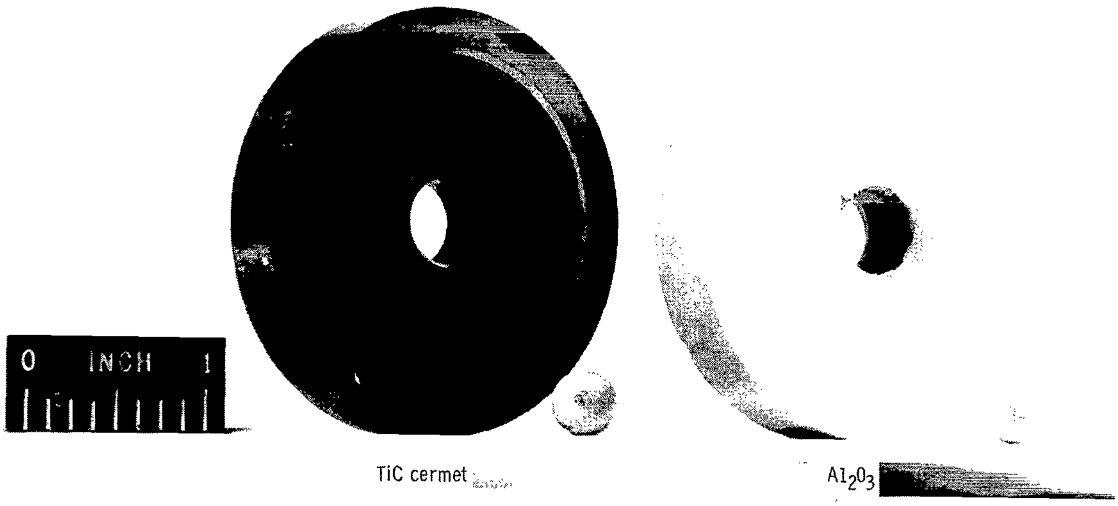
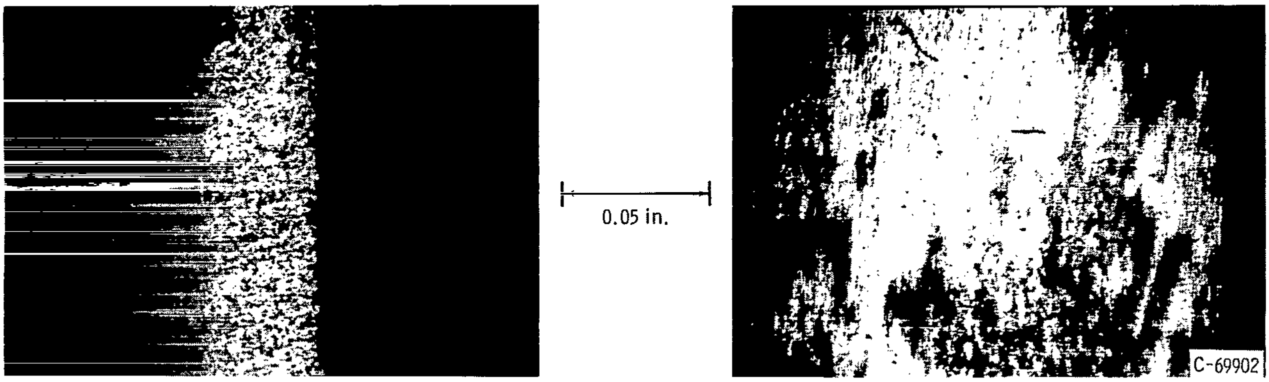


Figure 4. - Aluminum oxide riders and uncoated titanium carbide cermet and aluminum oxide disk specimens run submerged in liquid fluorine. Sliding velocity, 2300 feet per minute; load, 1000 grams.

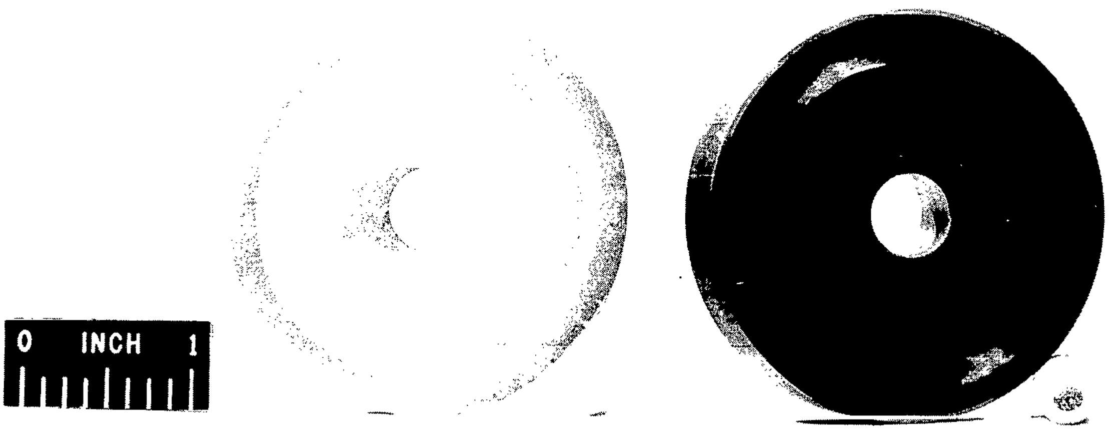
of the TiC cermet disk after it was in liquid fluorine. This indicated that the soft nickel binder reacted with the fluorine to form a thin surface film of NiF_2, which was responsible for the low coefficient of friction. Although AlF_3 was not positively identified on the Al_2O_3 disk run in fluorine, it is probable that a thin AlF_3 film was formed during sliding and was responsible for friction and wear characteristics.

Visual and microscopic examinations of the disk specimens run in oxygen and in fluorine show that the wear tracks are smooth and polished. The highly polished appearance of the TiC cermet suggests that the soft intermediate films, formed during sliding, prevented galling. The wear of the TiC cermet disk, therefore, is thought to be the removal of the soft nickel binder in the form of oxides or fluorides. Since profilometer traces of the Al_2O_3 disks indicated no appreciable wear in liquid fluorine, it is thought that the rider tip, being continually subjected to high interface temperatures, contributed



Disk wear area, Al_2O_3 (CaF_2 - LiF - NiF_2)

Disk wear area, nickel-chrome alloy (BaF_2 - CaF_2, eutectic)



Al_2O_3

Nickel-chrome alloy

C-67830

Figure 5. - Aluminum oxide riders and coated aluminum oxide and nickel-chrome disk specimens run submerged in liquid oxygen. Sliding velocity, 2300 feet per minute; load, 1000 grams.

most to the formation of the fluoride film. Because of its performance, both in liquid oxygen and in fluorine, the combination of Al_2O_3 sliding against the TiC cermet was selected as one of the material combinations for the fluorine seal phase of this investigation.

Friction and Wear with Applied Surface Films

The results of the applied fluoride coatings on either Al_2O_3 or the nickel-chrome alloy indicated that these coatings show promise in oxygen and in fluorine (table I and fig. 2). In general, the experiments showed the films performed at least as well in fluorine as they did in oxygen.

The CaF_2 + LiF + NiF_2 on Al_2O_3 appears capable of providing an effective mating surface for the Al_2O_3 rider in oxygen. When the results of this coat-

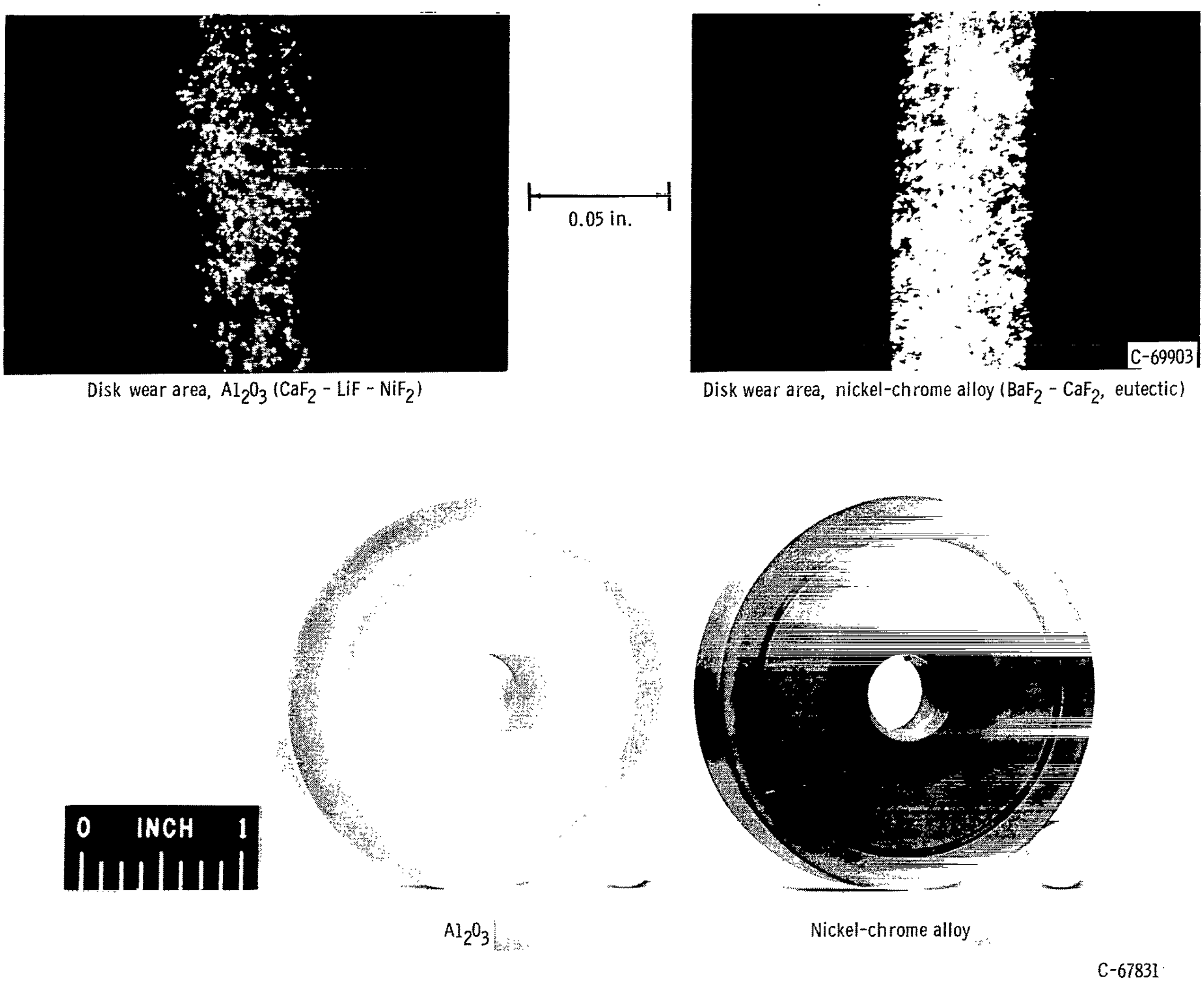


Figure 6. - Aluminum oxide riders and coated aluminum oxide and nickel-chrome disk specimens run submerged in liquid fluorine. Sliding velocity, 2300 feet per minute; load, 1000 grams.

ing are compared with the results of the uncoated Al_2O_3 (fig. 1) run in oxygen, it is noted that the friction coefficient was reduced from 0.50 to 0.29 by the coating. In fluorine, however, this fluoride coating on Al_2O_3 showed a higher friction coefficient (0.17) than the uncoated Al_2O_3 (0.12). Continuous friction traces recorded during runs in both oxygen and fluorine indicated that sliding was smooth and steady with the coating.

The Al_2O_3 rider wear in oxygen and in fluorine is essentially the same, and, although there is evidence of apparent coating failure (figs. 5 and 6), surface profile traces of the disk wear tracks show the wear of the Al_2O_3 substrate to be negligible. Because of the performance shown in the friction and wear experiments both in oxygen and in fluorine the CaF_2 + LiF + NiF_2 on Al_2O_3 was selected as a mating material for the Al_2O_3 nosepiece for the fluorine seal studies.

The applied film of $BaF_2 + CaF_2$ on the nickel chrome showed a great variance in performance when comparing the runs in oxygen and in fluorine. The runs in oxygen indicated that the film failed immediately and resulted in a deep grooving of the nickel-chrome substrate (fig. 5) as well as excessive Al_2O_3 rider wear (table I and fig. 2); the rider also showed evidence of metal transfer. The friction coefficient for this run remained high, and the recorded friction trace indicated very rough sliding and erratic contact.

In liquid fluorine, however, disk and rider wear are appreciably less (table I and fig. 2). Surface profile traces indicated very little wear to the nickel-chrome substrate. It is quite possible that NiF_2 was formed during sliding as was the case for the TiC cermet run in fluorine; however, X-ray diffraction did not detect its presence. The reduction in rider wear and disk wear and the low friction coefficient (0.24) are thought to be due to the effectiveness of the applied eutectic fluoride coating. Photographs of this combination after running in liquid fluorine are shown in figure 6.

Seal Experiments

From the friction and wear study, the material combinations of Al_2O_3 sliding against the TiC cermet and Al_2O_3 sliding against a fused coating (CaF_2 + LiF + NiF_2) on Al_2O_3 were selected as the materials to be used in the dynamic seal experiments in liquid fluorine (table II).

The stationary, flame-sprayed Al_2O_3 nosepiece was attached to a machined beryllium copper bellows that had a spring rate of 1000 pounds per inch and was run against the mating disk while submerged in liquid fluorine. The required experimental test conditions were as follows: sliding velocity, 2300 feet per minute; face load, 15 pounds; pressure differential across the seal, 2 pounds per square inch. In these seal experiments, the low pressure differential did not prevent the sealing surfaces from being wetted by liquid fluorine.

Two minor modifications to the friction and wear apparatus were required before the dynamic seal studies could be made (fig. 7). These modifications consisted in removing the loader assembly and redesigning the lower seal housing to incorporate the housing of the experimental seal. In addition, a helium purge line was connected between the load beam and the seal housing. This helium purge line was used to pressurize internally the experimental seal and to maintain a 2-pound-per-square-inch pressure differential across the seal. The results of the seal studies are presented in table II and figure 8.

In the first attempt to run a full-scale seal in fluorine it was found that the desired surface speed could not be attained because of insufficient power to drive the shaft. The overloading of the drive unit was caused by the high (21-lb) face load on the seal. As a result, this experiment was terminated after 20 minutes. Surface profile traces of the TiC cermet disk

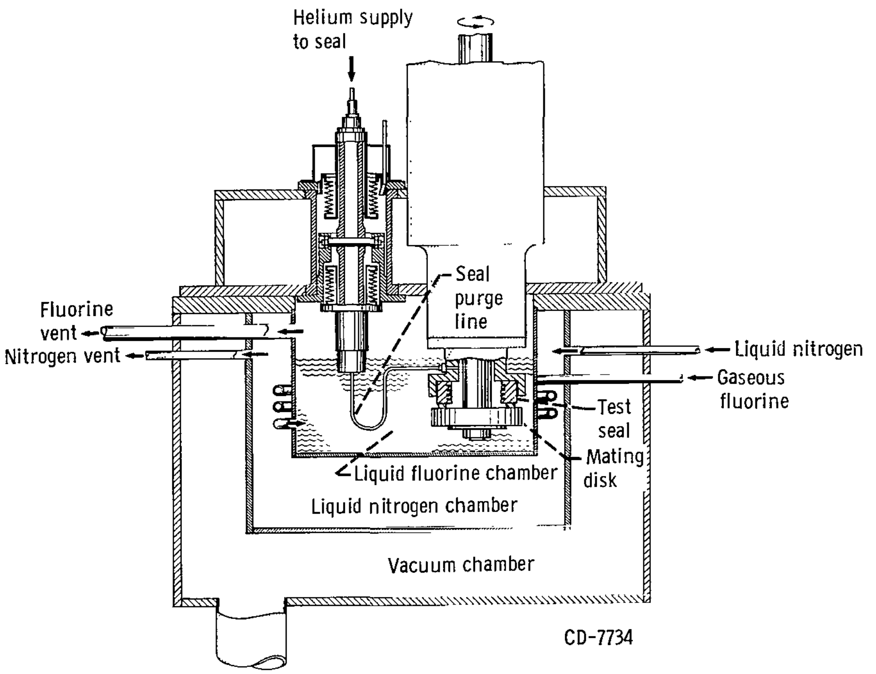


Figure 7. - Liquid fluorine seal test apparatus.

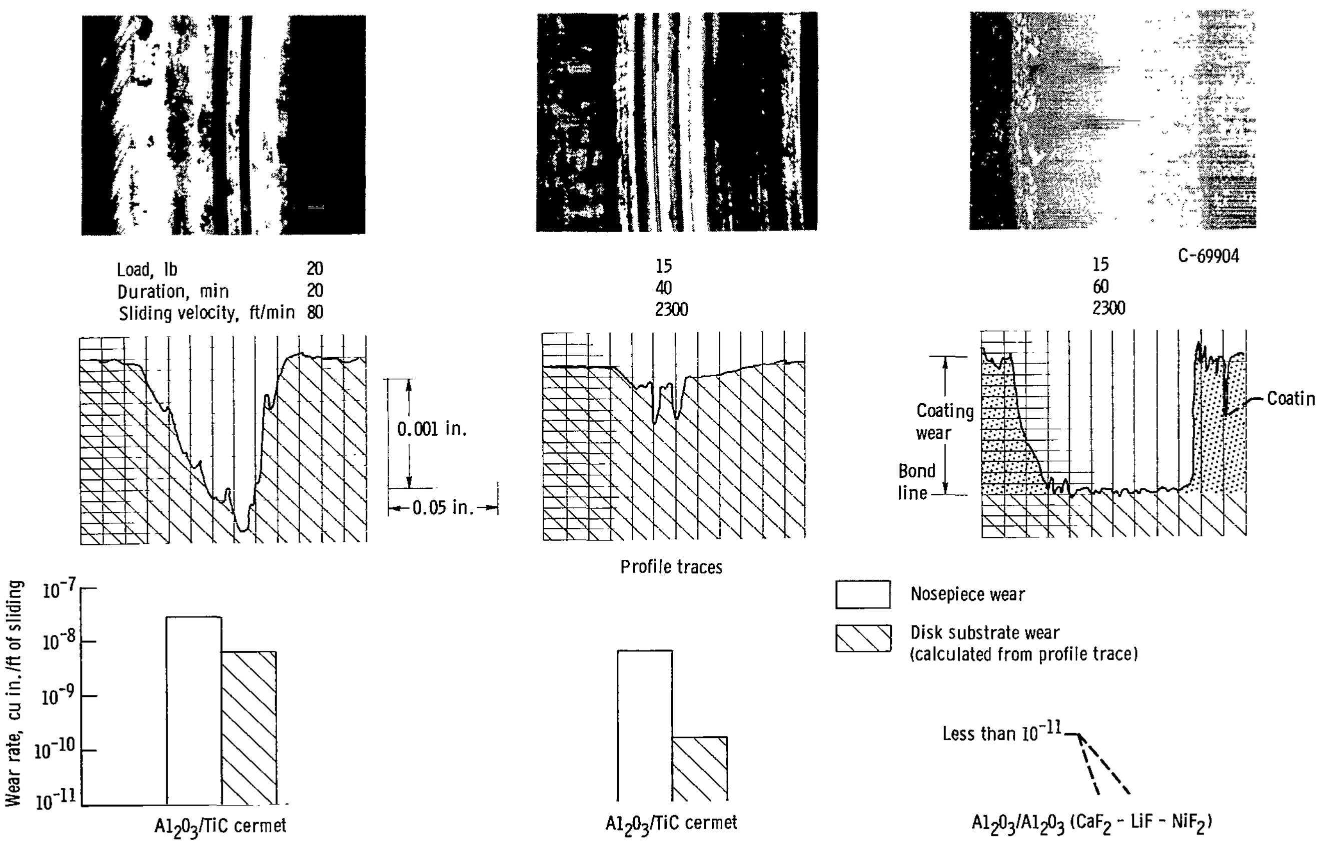


Figure 8. - Wear of seal materials obtained during seal experiments in liquid fluorine.

(fig. 8) indicated that a deep groove had been worn in the disk. The measurements of the Al_2O_3 nosepiece also indicated considerable wear in addition to visible edge chipping. Photographs of the seal components for this experiment can be seen in figure 9.

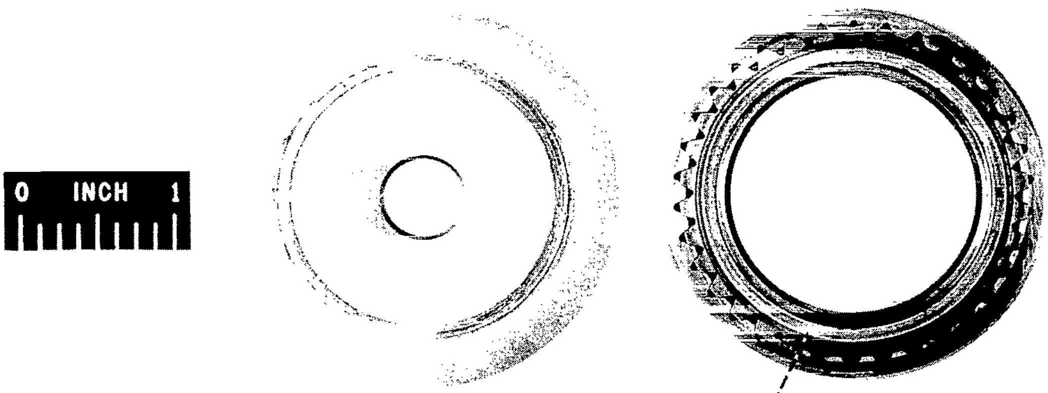


Figure 9. - Seal components; flame-sprayed aluminum oxide sliding against a titanium carbide cermet in liquid fluorine. Sliding velocity, 80 feet per minute; load, 21 pounds; duration, 20 minutes.

The second seal experiment was run after a modification to the drive unit was accomplished as well as calculations of expected seal face loading due to contraction of the shaft. This experiment was run successfully for 40 minutes at 2300 feet per minute and a face load of 15 pounds. Surface profile traces of the TiC cermet disk indicated a reduction in disk wear as compared with the first seal experiment (fig. 8). The Al_2O_3 nosepiece also showed less wear and less edge chipping. The seal components (fig. 10) are shown after completion of the experiment in fluorine.

The final seal experiment, that of Al_2O_3 running against a fused coating of CaF_2 + LiF + NiF_2 on Al_2O_3, was run successfully for 60 minutes. Visual examination of the mating disk indicated apparent loss of the film; however, X-ray diffraction showed that traces of the fused fluoride coating were present in the wear track. Surface profile traces of the mating disk indicated that there was no measurable wear of the Al_2O_3 substrate (fig. 8). Figure 11 shows the seal components used in the third seal experiment after 60 minutes of running in liquid fluorine. The results of the seal experiments indicated that Al_2O_3 sliding against either the fused fluoride film on Al_2O_3 or the TiC cermet are acceptable material combinations for fluorine seal applications. Proper pressure balancing of the seal is required, however, to prevent excessive face loading, which can cause catastrophic seal wear and local fragmentation of Al_2O_3.

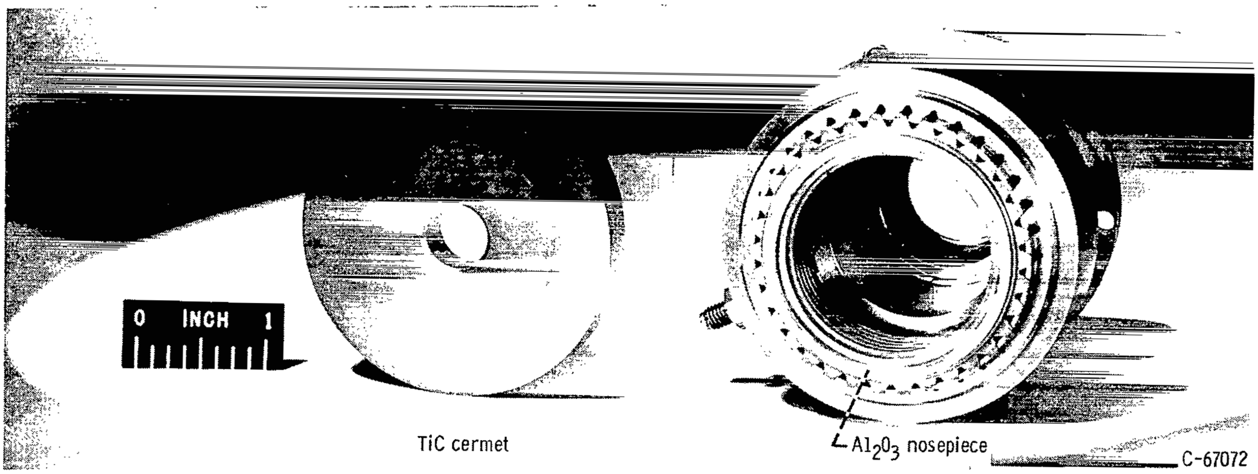


Figure 10. - Seal components; flame-sprayed aluminum oxide sliding against a titanium carbide cermet in liquid fluorine. Sliding velocity, 2300 feet per minute; load, 15 pounds; duration, 40 minutes.

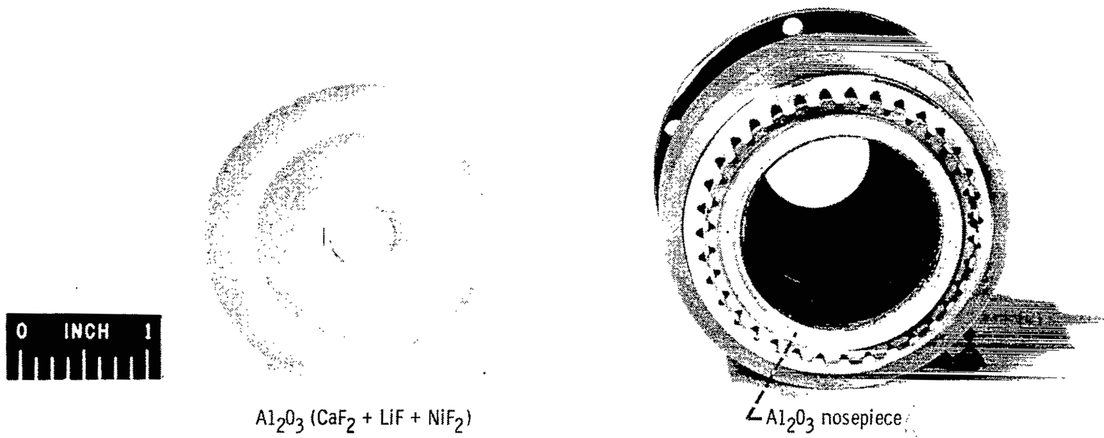


Figure 11. - Seal components; flamed-sprayed aluminum oxide sliding against 76 CaF_2 + 23 LiF + 1 NiF_2 on aluminum oxide in liquid fluorine. Sliding velocity, 2300 feet per minute; load, 15 pounds; duration, 60 minutes.

SUMMARY OF RESULTS

From the data obtained with aluminum oxide (Al_2O_3) in sliding contact with several mating surfaces submerged in liquid oxygen or in liquid fluorine in friction, wear, and dynamic seal studies, the following results were noted:

1. The Al_2O_3 sliding against either the titanium carbide cermet or a fused fluoride film on Al_2O_3 are potentially useful seal material combinations for liquid fluorine turbopump applications.

2. The presence of a fluoride film, either as an applied film (calcium fluoride (CaF_2) - lithium fluoride (LiF) - nickel fluoride (NiF_2)) or a film formed during sliding (NiF_2) is beneficial in reducing friction and wear of

materials in liquid fluorine.

3. With the same sliding combination in this investigation, the coefficient of friction was generally lower in liquid fluorine than in liquid oxygen.

Lewis Research Center
National Aeronautics and Space Administration
Cleveland, Ohio, June 4, 1964

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